Impact of post-depositional processes on charcoal fragmentation and archaeobotanical implications: Experimental approach combining charcoal analysis and biomechanics

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Generally speaking, charcoal analysis is based on identifying and counting charcoal fragments in order to calculate the relative variations in taxa frequency. All post-depositional processes are likely to induce fragmentation of the anthracological material, raising the question of the representativeness of taxa. Based on an innovative experimental approach combining both charcoal analysis and biomechanics, this paper explores how the mechanical properties of charcoal can influence the fragmentation and the quantification of species in anthracological assemblages. We carried out standardized laboratory compression tests on 302 samples issued from 10 taxa, charred at three different temperatures, in order to characterize the mechanical properties of common species in temperate and Mediterranean Europe. Our results highlight the differential responses of the tested species in terms of resistance to compression and fragmentation, two processes which do not appear to be correlated. Charcoal is very resistant to pressure (up to 22.5 MPa). Our results show that significant fragmentation differences exist between taxa. The total number of fragments after compression is largely dependent on the species, regardless of the charring temperature. However, this interspecific variability is more significant for small fragments [1–2 mm], than for larger fragments [2–4 mm] and >4 mm, with the exception of *Quercus*, which displays differential reactions to compression. Finally, a multifactorial analysis brings to light the impact of the physical and anatomical characteristics of the different species on charcoal fragmentation.

Keywords:
Charcoal analysis
Taphonomy
Mechanical properties
Fragmentation
Experimentation

1. Introduction

Charcoal preservation in archaeological sites is variable, even at sites where combustion structures have been identified (Théry-Parisot, 2001, 2010c). According to the current state of knowledge, there is no apparent relationship between charcoal preservation and the sedimentary context. Although this observation does not follow a chronological pattern, it is particularly true for Palaeolithic sites (Braadbaart et al., 2009, 2012; Beresford-Jones et al., 2010; Théry-Parisot et al., 2010a), where identifiable, macroscopic-sized charcoal is at times very rare and/or very altered. In such contexts, charcoal is primarily preserved among the mesoscopic and microscopic unidentifiable fraction (Marquer et al., 2010, 2012), raising the question of the impact of post-depositional processes on charcoal preservation. What are the effects of these processes on anthracological assemblages? Do they have a linear effect on charcoal or are some taxa more fragile than others due to their anatomical structure or chemical composition? This naturally brings us to the question of the representativeness of anthracological assemblages for assessing past vegetation and fuel management.

Since the end of the 1990s, some researchers have focused on a methodical approach to the formation process of anthracological assemblages (Théry-Parisot 1998, 2001; 2013; Théry-Parisot et al. 2010a,b), including (i) human practices, (ii) the physical and chemical modifications of wood during combustion, (iii) depositional and post-depositional processes. Human practices are dependent on non-predictable cultural factors specific to each group. On the other hand, combustion and post-depositional processes involve physico-chemical and mechanical transformations, which are independent of socio-cultural contexts, and which affect anthracological assemblages in terms of mass reduction and fragmentation. However, most palaeoenvironmental interpretations are partly based on variations in the relative frequency of taxa, calculated by counting the identified fragments in the studied sample. The effects of combustion on charcoal fragmentation have been the subject of numerous studies (cf. infra). Conversely, the effects of post-depositional processes on anthracological assemblages are not so well documented. However, all processes, from trampling to combustion residue, displacement by humans, weathering, water run off transport, bio or cryoturbation phenomena, alternating freeze-thaw cycles or sediment soaking-desiccation, can induce charcoal fragmentation (for a better overview of sites formation processes see for example Goldberg and Macphail, 2013). In this paper, we address the effects of depositional and post-depositional processes and the resulting potential modification of the assemblages.

The aim of this article is to characterize the physical properties of several common temperate and Mediterranean European species in the laboratory, using appropriate measurements on present-day material. By assessing the differential reaction of each species to mechanical post-depositional alteration processes, we can evaluate the palaeoecological representativeness of the anthracological assemblages. This study involves 302 samples issued from 10 taxa, charred at 3 different temperatures and subjected to standardized compression tests. The results should allow us to assess (i) the question of the mechanical properties of charcoal, (ii) the fragmentation level/rate of charcoal from different taxa and (iii) the modalities of this fragmentation. The main objective of this study is to produce data exposing the intrinsic mechanics of the tested species, to evaluate their conservation potential and to appraise the possibility of the under or over-representation of certain species in archaeological contexts.

2. Research history in charcoal fragmentation

After some pioneering studies (Salisbury and Jane, 1940; Santa, 1961; Vernet, 1973; Thiébault, 1980), methodological reflections on the representativeness of anthracological assemblages began in the 1980s (Chabal, 1988, 1997, 1990; Badal-Garcia, 1990, 1992). The first approach consisted in analyzing the global fragmentation rate and the size of the fragments of the different taxa in archaeological samples. The analysis of Protohistoric archaeological levels (Le Marduel, Lattes, south of France) yielded fragmentation histograms with a Poisson distribution for all the taxa within the same layer. These studies led to the formulation of the "single fragmentation law" hypothesis for all species (Chabal, 1991, 1997). "A posteriori" analysis included the undifferentiated fragmentation stages: combustion, post-depositional processes, sampling and sieving of the material. At the same time, several studies concentrated on the effects of combustion on anatomy and fragmentation (Rossen and Olson, 1985; Smart and Hoffman, 1988; Scott and Jones 1991; Prior and Gasson, 1993; Loreau, 1994; Vaughan and Nichols 1995; Belcher et al. 2005; Lingens et al. 2005; Braadbaart and Poole, 2008; Théry-Parisot and Chabel, 2010). These works demonstrated "the non-linearity of species behaviour towards fire suggesting that combustion is a taphonomic agent, which randomly affects deposits and whose effect on the assemblage is almost impossible to control" (Théry-Parisot et al., 2010a,b). The impact of post-depositional processes on anthracological assemblages is a less-developed aspect of research. Bio-turbation and transport were mainly studied in the domain of pedo-anthracology (Thinon, 1992; Vaughan and Nichols, 1995; Carcaillet and Talon, 1996; Blackford, 2000; Nichols et al., 2000; Scott et al., 2000; Scott, 2010; Carcaillet, 2001). Recent work on the impact of pH and diagenesis on the anthracological material has brought to light a structural alteration of charcoal in alkaline environments (Schiegl et al., 1996; Cohen-Ofri et al., 2006; Rebollo et al., 2008; Braadbaart et al., 2009; Ascough et al., 2010, 2011a,b), but also the strong influence of physical processes on the deterioration of the material (Braadbaart et al., 2009). Among these physical processes, mechanical actions, which directly influence the fragmentation of charcoal, play a preponderant role. Freeze/thaw laboratory experiments, coupled with measuring resistance to compression, have shown that wood alteration ante combustion has a strong incidence on the mechanical resistance of charcoal (Théry-Parisot, 1998, 2001). More recently, a study of the mechanical properties of species from the north of India showed that resistance to compression and the dimensions of the ensuing fragments is correlated to charring temperatures (Lancelotti et al., 2010). Mechanical resistance to compression is higher when compression is applied lengthwise to the cross-section and dense wood is more brittle than less compact woods. In temperate regions, abundant data are available concerning the properties of wood used for construction (Ashby, 2005; Forest Products Laboratory, 2010), but nothing indicates that these data can be transposed to charcoal. For this reason we developed an experimental study of the mechanical behaviour of present-day charcoal to evaluate the fragmentation process of the main species identified in temperate European anthracological assemblages.

3. Materials and methods

The mechanical tests were carried out in the École des Mines de Paris (ParisTech CEMEF- Sophia-Antipolis, France). The aim of these tests was to measure the mechanical response of charcoal to compression and the fragmentation modalities (number and size of fragments). The experimental procedure is based on previous work by Théry-Parisot (1998, 2001)

3.1. Sample preparation

Sample preparation must take account of both the (i) constraints of the shape of the samples used for the mechanical tests and (ii) the effect of the physico-chemical alteration on the mechanical properties of the material during combustion. It generally involves some form of standardization far removed from the reality of the studied archaeological contexts. The samples have to present two perfectly cut parallel sides with no structural, fissure type alteration.

The production of 2 cm cubes, with no charring fissures, is one of the restrictions of our protocol.

The tests concerned charred samples from 10 taxa commonly found in archaeological contexts in southern Europe: *Acer pseudoplatanus*, *Betula pubescens*, *Carpinus betulus*, *Corylus avellana*, *Fagus sylvatica*, *Fraxinus excelsior*, *Populus tremula*, *Pinus pinaster*, *Pinus sylvestris* and *Quercus pubescens*. The dry wood (12% moisture content) is issued from branches with a 10–15 cm section.

3.2. Charring protocol

In order to limit sample deformation during charring, each cube was wrapped in aluminium foil, placed in a porcelain crucible, covered with sand, and then charred in a muffle furnace. The charring temperature has a direct incidence on the mechanical resistance of charcoal (Hillis, 1984; Fengel and Wegener, 1989; Yildiz et al., 2006; Borrega and Kärenlampi, 2008; Gündüz et al., 2008; Mburu et al., 2008; Korkut et al., 2008; Korkut and

Hiziroglu, 2009; Kocaefe et al., 2010; Majano-Majano et al., 2012; Poletto et al., 2012). Consequently, the choice of temperature must take into consideration the different stages of the thermal deterioration of the wood (Byrne and Nagle, 1997; Rousset et al., 2006; Braadbaart and Poole, 2008), but must also reflect the probable temperatures of archaeological hearths (Costamagno et al., 2010; Théry-Parisot and Chabal, 2010). The samples were charred at three different temperatures: 400, 500 and 750 °C, in porcelain crucibles placed in the hot oven during 30 min. 302 of the 400 prepared samples were usable, representing a 25% loss principally for samples charred at 400° and 750 °C. On average, 30 samples were tested for each species, with an average of 9.1, 15.6 and 5.5 samples at temperatures of 400 °C, 500 °C and 750 °C respectively.

3.3. Physical characterization

The physical properties of each sample were recorded in order to evaluate their incidence on the fragmentation process. The mass (M for dry wood and MC for charcoal), and the dimensions of the cube were measured in 3 directions, first on air-dried wood and then on charcoal. This allowed for the calculation of the volume (V for dry wood and VC for charcoal), the density using the formula $D = M/V$ (WD for dry wood, WCD for charcoal), mass loss (M − MC)/M (Mloss %), volume loss (V − VC)/V (Vloss %) and density loss (WD − WCD)/WD (Dloss %).

Systematic porosity measurements were taken on the transverse sections of charcoal samples using SEM image analysis. The porosity ratio corresponds to the difference in porosity between the initial and final wood. These measurements were incorporated into the multivariate analysis to include the impact of the porosity of taxa on their mechanical properties and fragmentation. A macro for this measurement was written with image J software (Ducom, 2010).

3.4. Compression procedure

A hydraulic traction-compression testing machine, equipped with a 10 kN sensor (Instron 1121), was used for the compression experiments. Pressure was applied parallel to the fibers. The descent speed was fixed at 0.1 mm/s during 30 s from the time of contact with the sample, which was placed on its transversal side.

The pressure applied and the resulting decrease in the height of the sample (displacement of the tray), were recorded during the tests. These values allowed us to plot the stress (MPa)/strain (%) curves, by dividing the force by the surface of the transversal side of the sample (stress), on one hand, and the displacement by the height of the sample (strain), on the other hand.

Fig. 1 represents a typical test curve. It is comprised of three main parts:

- a gradual rise after the initial contact, which corresponds to setting up the compression;
- a rapid and linear rise of the stress until the peak, which corresponds to the crushing of the whole sample;
- a more or less brutal decrease in stress, possibly with non-negligible drag strain, corresponding to the destruction of the charcoal structure.

It is noteworthy that compression is never reversible; it is not therefore an elastic phenomenon.

This diagram allows us to calculate several mechanical indicators in phase 2:

- Maximum stress called crushing strength (CS in MPa);
- Crushing modulus (CM in MPa);

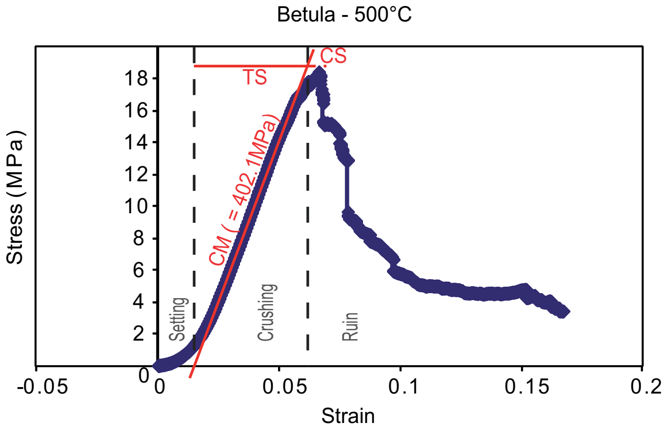

Fig. 1. Typical stress–strain curve of the compression test (comprising the three main phases: setting up the compression test, crushing and destruction of the charcoal structure) and mechanical indicators calculated (CS, CM and TS).

- Theoretical crushing strain (TS = CS/CM).

Crushing strength (CS) is directly linked to the maximum force (pressure) that can be applied to a piece of charcoal before complete destruction. If the CS is low, the charcoal is less resistant to pressure.

Theoretical crushing strain (TS) is mainly related to the time required to completely crush the charcoal. If TS is low, the charcoal is more fragile in the compressive test.

Crushing modulus (CM), like any modulus, relates charcoal contraction to the compressive force exerted on it. If CM is low, the charcoal is less rigid during compression.

As both wood and charcoal are honeycomb-like materials, mechanical properties in grain direction are roughly proportional to their density (Gibson and Ashby, 1999). It is therefore interesting to use "specific" crushing strength, i.e. CS divided by charcoal density: CS/WCD (expressed in 10^3 m^2/s^2), as a criterion, independently of density itself.

The applied pressure induces sample fragmentation. The residues of each test were systematically sieved in three different meshes: [1–2 mm], [2–4 mm], > 4 mm and the fragments were then counted using image analysis (ImageJ).

3.5. Statistical methods

Several univariate to multivariate statistical methods were used to analyze the data and to assess the relation between the mechanical properties and the fragmentation process. The choice of tests depends on the purpose of the analysis and the nature of the available data. Non-parametric tests based on the ranks of the values were chosen when the normality of the distribution was not verified. The Spearman's rank correlation coefficient is a nonparametric measure of statistical dependence between two variables. Regression analysis was used when the dependent variable follows a Normal distribution. The very flexible and extremely powerful one-way analysis of variance (ANOVA), widely used for the analysis of experimental schemes, can simultaneously analyse several scenarios and detect the effects of independent nominal variables on a large number of continuous variables. This test was performed to assess and evaluate the effect of the charring temperature and the taxon on the fragmentation process. The Bonferroni–Dunn test allows for comparisons, controlling the family error rate. It was carried out in order to identify groups of taxon with the same fragmentation process. A multiple correspondence analysis (MCA) was applied to our data set categories to test relationships between variables.

4. Results (Table 1)

4.1. Physical and mechanical properties

4.1.1. Mass, volume and density

In our tests, charring induces severe mass, volume and density loss.

If we disregard wood water loss at the beginning of charring (on average 12%), on average, mass loss (Mloss) approaches 55% at 400 °C, 60% at 500 °C and 70% at 750 °C. There are no systematic significant differences between the different species.

Volume loss (Vloss) is about 50% at 400 and 500 °C and increases to 58% at 750 °C, which is considerable in comparison to the hygroscopic shrinkage between air-dried wood and oven-dried wood (about 6–8%). In this case, there are noticeable systematic differences between species, with *Quercus pubescens* displaying the lowest volume shrinkage and *Carpinus betulus* the highest.

Density loss (Dloss) increases markedly with temperature (Fig. 2a). It rises from 32% at 400 °C, to 40% at 500 °C, and reaches 55% at 750 °C. Average density loss is 40%, with variations from 33 to 50% according to the species (Fig. 2b), with *Quercus* and *Carpinus* representing the two extremes (*Quercus* records a much higher density loss than the other species – which tends to slot it into the average – whereas that of *Carpinus* is much lower).

Charring tends to regulate density differences between species but charcoal density remains globally proportional to the initial wood density (Fig. 2c). The Spearman correlation test indicates a significant positive correlation between the two series of values (rho = 0.69/*p*-value < 2.2^{e}−16) (Table 2).

4.1.2. Crushing strength (CS), crushing modulus (CM) and theoretical crushing strain (TS) (Fig. 3)

- The effect of temperature

The three mechanical indicators evolve markedly with temperature (Table 1). All three decrease when the charring temperature increases, with crushing strength (CS) being more affected than the others. The crushing modulus (CM) decreases slightly between 400 °C (424 MPa) and 500 °C (396 MPa), but is almost halved at 750 °C (220 MPa) (Fig. 3a). Crushing strength (CS) varies on average from 16.9 MPa at 400 °C, to 14.5 MPa at 500 °C and 6.9 MPa at 750 °C (Fig. 3b). As for the theoretical crushing strain (TS), it decreases regularly from 4.3% at 400 °C to 3.2% at 750 °C, with a value of 3.8% at 500 °C (Fig. 3c).

It appears from these results that charcoal becomes less resistant and more fragile as charring temperatures increase.

Table 1
Average values for mechanical properties and fragmentation of the 10 species and 3 heat treatments. Abbreviations: Nb: number of samples; WD: wood density; WCD: charcoal density; Mloss: mass loss; Vloss: volume loss; Dloss: density loss; >4 mm, 2–4 mm, 1–2 mm: number of fragments in each class size; Total: total number of fragments; CM: crushing modulus; CS: crushing strength; TS: theoretical crushing strain; CS/WCD: specific crushing modulus.

Species	T°	Nb	WD	WCD	Mloss	Vloss	Dloss	>4 mm	2–4 mm	1–2 mm	Total	CM	CS	TS	CS/WCD	Means	
																CS	CM
Acer	400	9	0.65	0.41	0.68	0.49	0.37	20	52	82	154	461	19.8	0.044	48.6		
	500	16	0.65	0.39	0.71	0.51	0.40	19	66	105	189	437	19.9	0.046	51.0	18.9	429
	750	4	0.64	0.28	0.81	0.58	0.54	19	61	87	167	324	13	0.038	42		
Betula	400	9	0.59	0.40	0.69	0.55	0.32	17	34	96	147	501	20.4	0.041	51.3		
	500	15	0.59	0.36	0.74	0.56	0.39	15	41	136	191	426	16.7	0.039	46.1	16.1	407
	750	5	0.60	0.27	0.84	0.64	0.55	17	33	106	156	181	6.9	0.037	25.5		
Carpinus	400	11	0.73	0.54	0.70	0.59	0.26	20	45	156	221	548	20.6	0.042	38.1		
	500	15	0.73	0.49	0.74	0.62	0.33	16	44	160	220	591	19.9	0.033	40.9	19.4	557
	750	2	0.74	0.44	0.82	0.68	0.42	15	40	114	169	356	9.5	0.030	22.3		
Corylus	400	7	0.60	0.43	0.68	0.55	0.28	17	48	137	202	475	22.5	0.049	51.7		
	500	16	0.63	0.40	0.73	0.57	0.37	16	54	156	227	445	20.3	0.047	51.3	17.2	394
	750	7	0.65	0.29	0.83	0.60	0.56	17	43	109	169	198	4.9	0.026	16.8		
Fagus	400	8	0.69	0.50	0.66	0.53	0.28	16	37	62	115	408	15.3	0.041	30.7		
	500	16	0.68	0.41	0.71	0.52	0.39	17	43	71	131	436	14.2	0.033	33.9	13.9	402
	750	4	0.69	0.33	0.81	0.58	0.53	17	44	69	129	258	9.6	0.037	28.3		
Fraxinus	400	11	0.72	0.52	0.60	0.45	0.27	20	47	141	207	437	12.9	0.031	24.7		
	500	14	0.74	0.45	0.71	0.52	0.40	16	64	210	290	406	12.4	0.034	27.8	11.8	390
	750	7	0.76	0.32	0.82	0.58	0.58	17	50	149	216	286	8.8	0.030	25.5		
Pinus pinaster	400	8	0.54	0.34	0.62	0.40	0.37	17	41	66	124	351	19.0	0.056	56.2		
	500	16	0.60	0.35	0.68	0.45	0.41	22	63	102	187	357	11.3	0.037	32.6	12.4	320
	750	6	0.52	0.23	0.80	0.55	0.56	22	58	78	157	181	6.4	0.037	27.8		
Pinus sylvestris	400	8	0.52	0.36	0.63	0.46	0.31	24	48	73	144	345	12.7	0.042	35.5		
	500	16	0.52	0.31	0.70	0.50	0.40	19	62	108	189	284	10.2	0.039	32.9	9.6	272
	750	7	0.54	0.23	0.82	0.56	0.58	18	58	100	176	164	4.6	0.028	19.6		
Populus	400	8	0.49	0.33	0.68	0.53	0.32	14	28	45	87	377	15.6	0.049	46.7		
	500	16	0.49	0.28	0.74	0.55	0.42	13	33	54	100	295	11.8	0.041	41.5	11.2	283
	750	7	0.48	0.20	0.83	0.58	0.58	19	42	60	121	138	4.1	0.029	19.8		
Quercus	400	8	0.91	0.51	0.63	0.34	0.44	40	75	139	255	337	10.4	0.033	20.8		
	500	16	0.93	0.46	0.66	0.31	0.50	35	88	144	267	285	7.8	0.028	17.1	8.1	281
	750	7	0.85	0.32	0.81	0.48	0.62	23	60	97	180	165	4.3	0.027	13.5		

T°	T°	Nb	WD	WCD	M loss	V_{loss}	D_{loss}	>4 mm	2–4 mm	1–2 mm	Total	CM	CS	TS	CS/WCD
Means	400	91	0.66	0.44	0.66	0.49	0.32	21	46	103	170	426	16.7	0.042	39.3
	500	155	0.66	0.39	0.71	0.51	0.40	19	56	124	200	394	14.4	0.038	37.2
	750	55	0.64	0.28	0.82	0.58	0.56	19	50	97	165	210	6.6	0.031	23.2

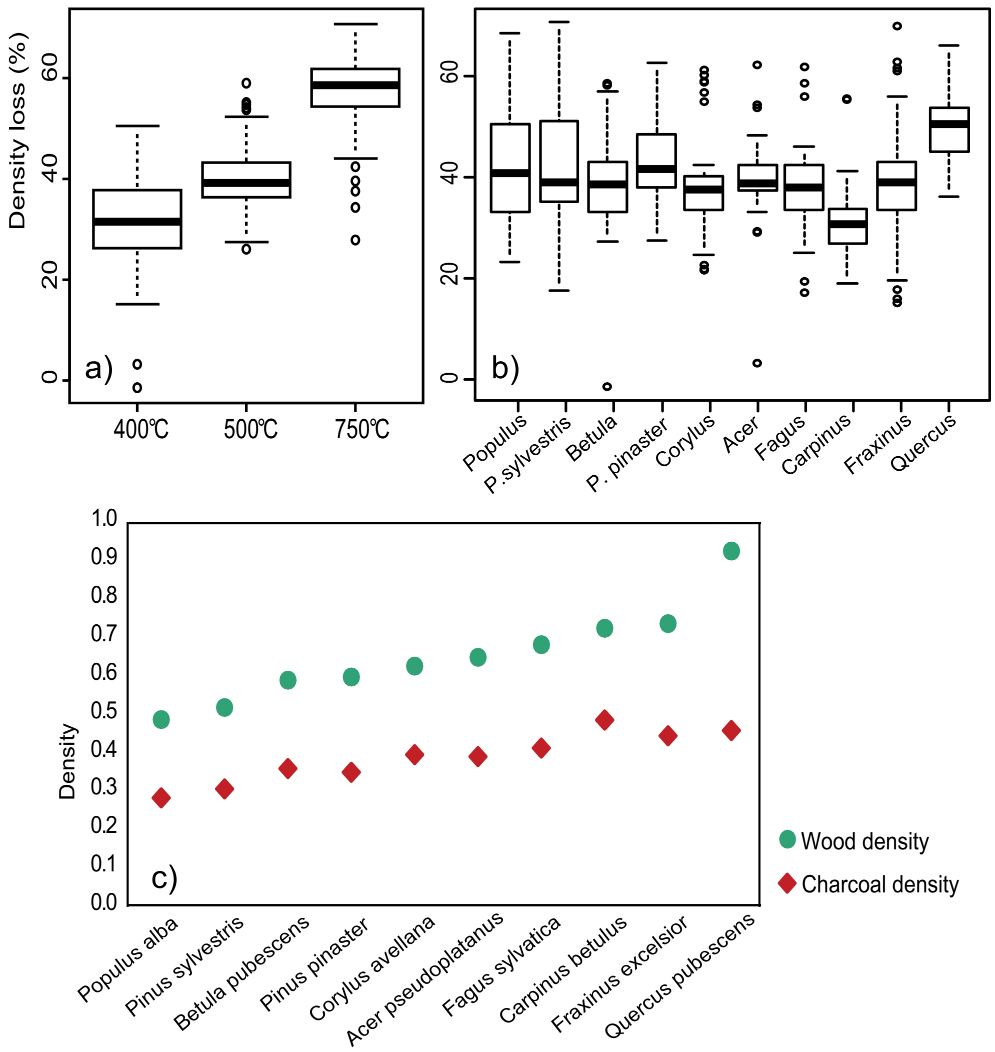

Fig. 2. Mean density loss by charring temperatures (a), by species (b), Mean density of wood and charcoal by species (c).

■ Species effect

Due to the fact that a larger and more constant number of specimens were charred at 500 °C, the mean values at this temperature were used to compare species.

The three indicators vary according to the different species (Table 1), which partly explains the substantial standard deviations observed for this temperature. The differences between taxa are noticeable, with CM ranging between 285 MPa for *Quercus* and 591 MPa for *Carpinus*, CS between 7.8 MPa for *Quercus* and 20.3 MPa for *Corylus* and TS between 2.8% for *Quercus* and 4.7% for *Corylus*. Quercus is by far the least resistant and most fragile taxon, while *Acer*, *Carpinus* and *Corylus* are the most resistant and the least fragile. The difference is even more accentuated between *Quercus* and *Corylus* for the specific crushing strength: respectively 17.1 and 51.3.

■ Interaction between the effects of temperature and species

An increase in temperature globally induces a decrease in charcoal resistance (CS), regardless of species (Fig. 3). This decrease is partly due to the reduction in charcoal density, which is most pronounced between 500 and 750 °C.

According to the CIRAD database (Cirad database TROPIX® 7, 2011), measurements taken on 243 air-dried wood samples from tree species, with densities ranging from 0.2 to 1.2, show that resistance to axial compression is proportional to the density of dry wood (WD): CS = 84.7 * WD, with a coefficient of regression $R^2 = 0.88$. The CS/WD ratio is called specific resistance (in relation to density) and varies little between species.

It is therefore interesting to compare the specific resistance of charcoal (CS/WCD) to the mean value observed for air-dried wood. On average (Table 1), the ratio between (CS/WCD) and this mean value (84.7) is still 0.48 and 0.44 for 400 °C and 500 °C respectively, but it drops to 0.28 at 750 °C.

There are marked differences in temperature between species when the sudden drop in resistance occurs (Fig. 4). *Fraxinus*, *Fagus* and *Quercus* already display low values at 400 °C and these values do not fall suddenly at 750 °C. *Pinus pinaster* decreases clearly between 400 and 500 °C but not so much between 500 and 750 °C. All the other species present a strong decrease in resistance between 500 and 750 °C.

4.2. Fragmentation

4.2.1. Global fragmentation process, all class sizes combined

After the compression tests, the samples produced on average, 170 fragments at 400 °C, 200 fragments at 500 °C and 165 fragments at 750 °C (Fig. 5a). The relationship between temperature

Table 2
Table of correlations between variables (Spearman test).

Pairs of variables		Coef. correlation (Rhô)	p-Value	Coef. determination
WD	WCD	0.708	<0.0001	0.501
Total Nb fragt	WD	0.517	<0.0001	0.267
Total Nb fragt	WCD	0.433	<0.0001	0.188
Total Nb fragt	CM	0.170	0.003	0.029
Total Nb fragt	CS	0.070	0.225	0.005
Total Nb fragt	CS/WCD	−0.090	0.120	0.008
Total Nb fragt	Dloss	0.019	0.739	0.000
Nb fragt [1–2 mm]	WD	0.496	<0.0001	0.246
Nb fragt [1–2 mm]	WCD	0.464	<0.0001	0.215
CM	WCD	0.516	<0.0001	0.266
CM	Dloss	−0.587	<0.0001	0.344
CS	WCD	0.404	<0.0001	0.163
CS	Dloss	−0.671	<0.0001	0.451

and the number of fragments is therefore not consistent during our tests. The total number of fragments for each species presents marked variability, ranging from 34 fragments for *Populus* to 400 for *Fraxinus*, with, on average, 101 fragments for *Populus* and 246 for *Quercus pubescens*.

The differences between species are significant, as shown by the results of the ANOVA (Table 3), which make it possible to classify the effects of temperature and species. Both factors combined account for 61.5% of the total variability but the *F*- value and the sum of the squares for the SPECIES factor are more significant, explaining 49.7% (R^2) of the variation of the model. The histogram (Fig. 6) summarizing the total number of fragments produced by compression, according to species and temperature, clearly shows the preponderant role of the taxon on fragmentation. Apart from some rare exceptions (500 °C/*Fraxinus*), the observed variability is much more significant between the different species than between temperatures.

A bilateral paired-comparison test brings to light four partly overlapping groups of taxa. *Populus* on one hand, and *Quercus* and *Fraxinus* on the other hand, display contrasting responses, which differ from those of most species. On average, *Populus* is not very fragmented whereas *Quercus* and *Fraxinus* are much more fragmented. The other species are clustered around two median groups. *Fagus* is similar to *Populus*, followed by a group made up of *P. pinaster*, *Acer* and *Betula*. *P. sylvestris*, *Corylus* and *Carpinus* tend to display slightly greater fragmentation and *P. sylvestris* has median fragmentation (Table 4).

4.2.2. Mechanical properties versus fragmentation

Series of Spearman tests show that, on the whole, there is virtually no link between mechanical properties and the fragmentation process (Table 2). Mass loss, crushing strength and specific crushing

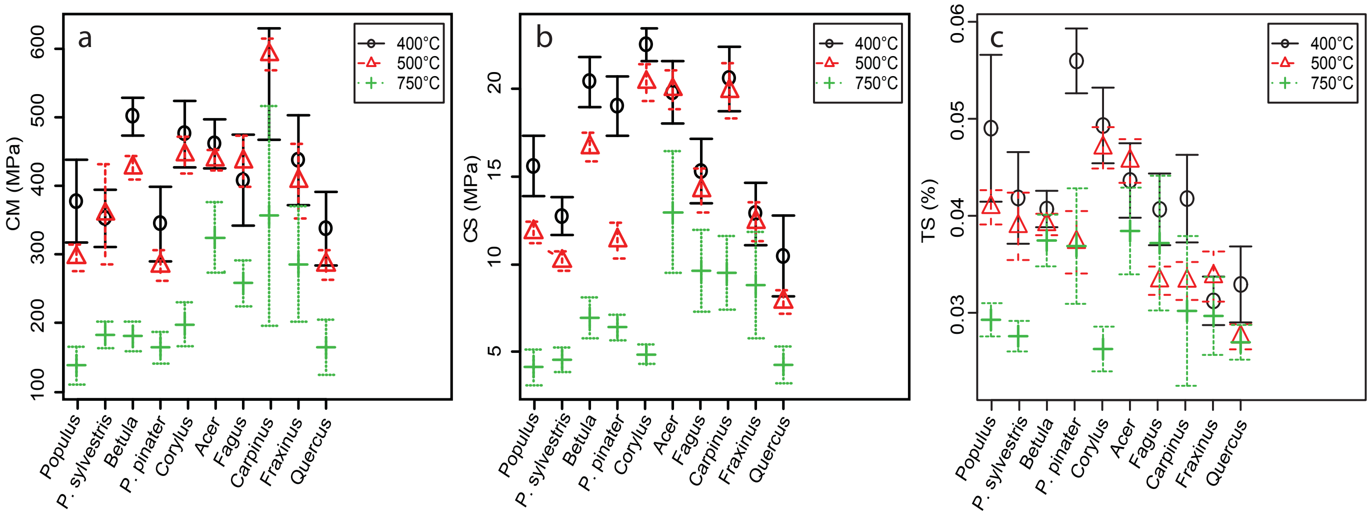

Fig. 3. Interaction between temperatures and species on the mechanical properties: crushing modulus CM (a); crushing strength CS (b); theoretical crushing strain TS (c).

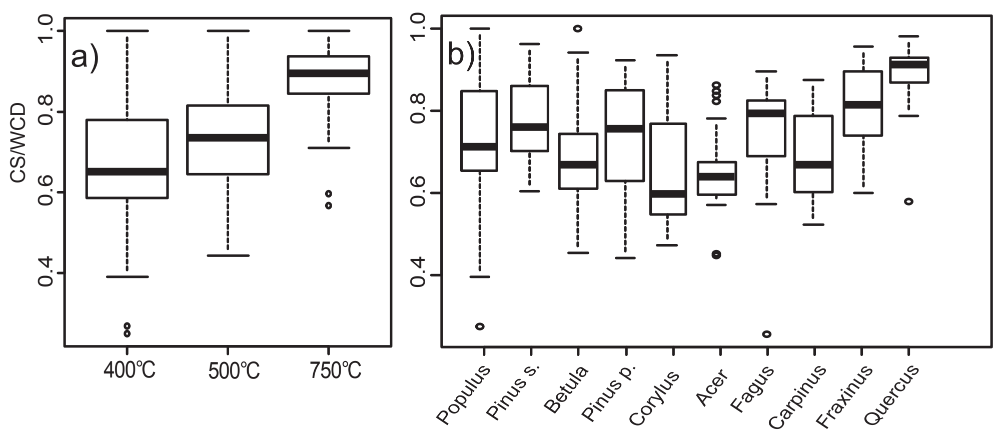

Fig. 4. Specific rupture strength (CS/WCD) by: temperature (a) and species (b).

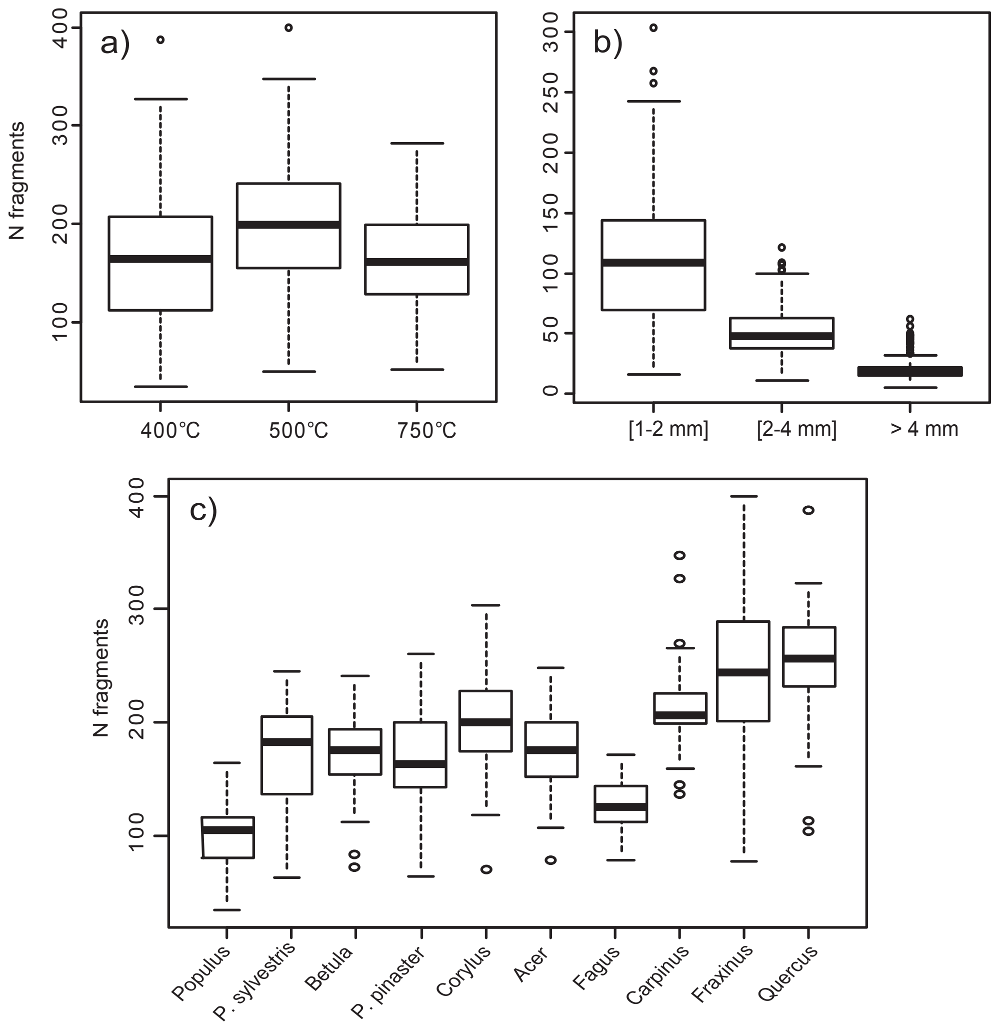

Fig. 5. Box & Whiskers plot for the total number of fragments: by charring temperature (a), by class sizes (b), by species (c).

strength are not correlated with the total number of fragments produced. The crushing modulus is very slightly correlated with the total number of fragments: the *p*-value is significant (0.0076), but the determination coefficient is low (rho = 0.15). Scatter graph (Fig. 7) explicitly shows that there is no relation between crushing strength (which is a good expression of the brittleness of charcoal) and the number of fragments produced by compression tests. In other words, mechanical properties and fragmentation are strongly dependent on the species, but there is no correlation between the two factors themselves. Each species is more or less resistant to compression but once the fragmentation process has begun, it affects each species independently of the pressure applied.

4.2.3. Fragmentation modalities by class size [1–2 mm], [2–4 mm], >4 mm

The first observation concerns the disparity in fragment distribution between the different class sizes. All species combined, the average number of fragments by class size is 111 in the smallest class size [1–2 mm], 51 in the second [2–4 mm], and only 19 fragments larger than 4 mm (Fig. 5b).

According to the species, the number of fragments varies from 16 to 304 in the class [1–2 mm], from 11 to 121 in the class [2–4 mm] and from 5 to 62 in the class >4 mm (Fig. 5c). The variance of the three series is thus quite marked and the coefficient of variation is around 40% for the three class sizes.

Fragmentation variability by species is very significant in the class [1–2 mm] with substantial tiering of the dispersion boxes (Fig. 8a). *Populus* is the least fragmented species with a minimum of 16 fragments and *Fraxinus* is the most fragmented species with a maximum of 304 fragments. The bilateral paired-comparison test (Bonferroni–Dunn procedure, Table 4) distinguishes four significantly different groups.

The upper and lower extremes are respectively made up of an isolated species, *Populus*, and a group made up of *Corylus*, *Quercus*, *Carpinus*, *Fraxinus*, which generally yields more fragments. *Fagus* and *P. pinaster* are similar to *Populus*, whereas *Acer*, *P. sylvestris* and *Betula* display intermediate fragmentation, between the species with high and low fragmentation. Variability is less marked in the intermediate class [2–4 mm], where divergence between the species is slightly reduced and only two species deviate: *Populus* generally produces less fragments (mean = 33) and *Quercus* (mean = 78) produces on average a lot more fragments. As for the other species, *Betula*, *Fagus*, *Carpinus* and *Corylus* present a low fragmentation tendency whereas *Fraxinus*, *P. Sylvestris*, *P. pinaster* and *Acer* display a high tendency (Fig. 8b).

Lastly, in the >4 mm class, the dispersion boxes have a more or less constant distribution, apart from *Quercus*, which is quite detached from the other species (Bonferroni–Dunn-test). Variability is generally much less significant here (Fig. 8c).

Table 3
Effect of *species* and *temperature* on fragmentation (ANOVA one-way variance analysis and PLSD Fisher test).

Variable	DDL	Sum of the squares	*F* value	Pr > *F*
Species	9	671386.8	40.762	<0.0001
Temperature	2	45851.4	25.054	<0.0001
Species * temperatures	18	68889.6	2.091	0.006

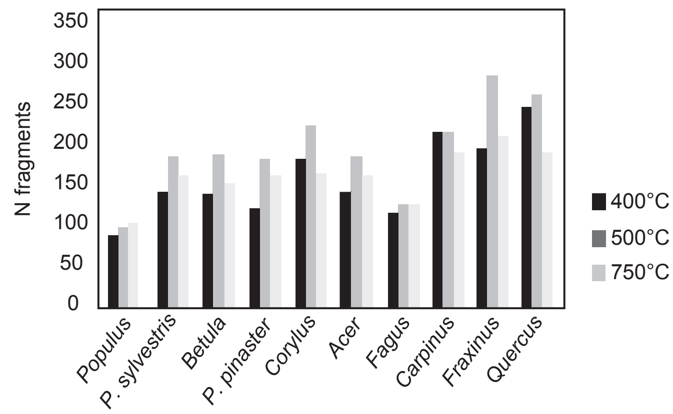

Fig. 6.

Differences between taxa are thus very slight in the >4 mm size and relatively minor in the [2–4 mm] class. They are most marked in the [1–2 mm] class.

4.3. Multivariate analysis

The multiple correspondence analysis (MCA) conducted on all the specimens charred at 500 °C allows us to discuss the relationships between our results and the main physical and anatomical characteristics of the studied species. Only the variables with a strong impact on the analysis have been retained (defined by two or three modalities: high/medium/low). For example, fiber thickness was initially tested and then removed. Moreover, the species themselves are integrated as supplementary variables and are not used for the calculation of the ACM (Fig. 9).

On axis 1, accounting for 62. 21% of the total variance, the “large vessels”, “very large and long rays” and “high porosity” modalities are closely linked. These modalities are secondarily associated with the absence of spiral thickenings and are related to a low crushing strength (CS) and a high number of fragments >4 mm. On axis 2, in the upper right part of the graphic, in relation to the active variables on axis 1, “high density” is strongly correlated with the high values of the total number of fragments and the high number of fragments >1 mm.

In the upper left part, the high crushing strength is strongly correlated to the presence of spiral thickenings and, secondarily, to the “small aggregate vessels” modality.

Lastly, in the lower part of the graphic, the “low density” (WCD) and “small isolated vessels or homoxylous wood” are linked to the low total number of fragments.

Wood with a marked porous zone, with large vessels, such as *Quercus* and *Fraxinus*, and with long radial file vessels (*Carpinus* and *Corylus*) are the most fragmented. Homogeneous wood (small isolated diffuse vessels or homoxylous) such as *Populus*, *Acer*, *Betula* or *P. pinaster*, *P. sylvestris* are those that produce fewer fragments. *Corylus*, *Carpinus* and *Acer* present a higher crushing strength than the other taxa. This characteristic seems to be linked to the presence of spiral thickenings. Conversely, the weak value of crushing strength is either linked to the porosity of the charcoal (case of *Fraxinus* and *Quercus*), or to its low density, as is the case for *Populus*, *P. sylvestris* and *P. pinaster*. In all cases, wood with a low density presents a low value for crushing strength. On the other hand, dense wood alternatively yields either very high or very low values.

4.4. Results synthesis

Table 3 summarizes correlation coefficients and brings to light the differential responses of the tested species as regards resistance to compression and fragmentation. These two processes are clearly not related. We have observed that:

- Temperature increase generally induces a decrease in the resistance of the charcoal, whatever the species (Fig. 3). This is similar to the results of tests carried out at low temperatures on industrial wood (Gündüz et al., 2008; Korkut et al., 2008; Korkut and Hiziroglu, 2009; Kocaefe et al., 2010; Majano-Majano et al., 2012). Crushing strength loss is particularly noticeable above 500 °C.
- Density loss is correlated to the charring temperature but is also related to the species. However, charring tends to attenuate the differences in density between species (Fig. 2).
- Density loss induces crushing strength loss and accounts for the main mechanical properties of charcoal.
- All temperatures combined, charcoal resistance is also linked to the species. The mechanical properties of the species are modified at distinctive thermal thresholds (Fig. 3).
- Resistance to pressure is dependent on the species but fragmentation intensity is not correlated to the resistance of the material itself. The least resistant species are not those subject to the most fragmentation (Fig. 7).
- The relationship between the number of fragments produced by compression and temperature is not constant (Fig. 5a).
- The total number of fragments after compression depends mainly on the species, regardless of temperature (Table 3 and

Table 4
Groups of species vs. fragmentation (Dunn–Bonferroni Test).

Total

Taxon	Groups				
Populus	A				
Fagus	A	B			
P. pinaster		B	C		
Betula		B	C	D	
Acer			C	D	
P. sylv.			C	D	
Corylus			C	D	E
Carpinus				D	E
Fraxinus					E
Quercus					E

[1–2 mm]

Taxon	Groups			
Populus	A			
Fagus	A	B		
P. sylv.	A	B	C	
Acer		B	C	
P. pinaster		B	C	
Betula			C	D
Corylus				D
Quercus				D
Carpinus				D
Fraxinus				D

[2–4 mm]

Taxon	Groups				
Populus	A				
Betula	A	B			
Fagus	A	B	C		
Carpinus	A	B	C	D	
Corylus		B	C	D	
Fraxinus			C	D	
P. pinaster			C	D	
P. sylv.			C	D	
Acer				D	E
Quercus					E

>4 mm

Taxon	Groups		
Populus	A		
Betula	A		
Corylus	A	B	
Fagus	A	B	
Carpinus	A	B	
Fraxinus	A	B	
P. sylv.	A	B	
Acer	A	B	
P. pinaster		B	
Quercus			C

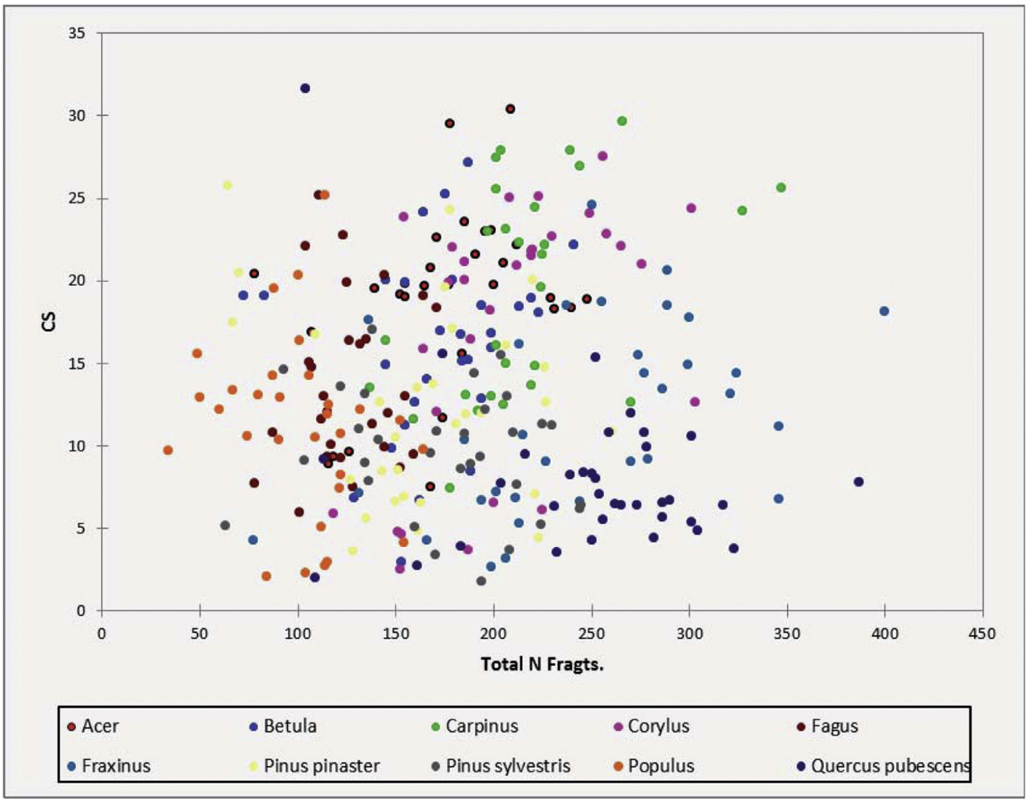

Fig. 7. Effect of the Specific rupture strength on the number of fragments.

Fig. 6). *Populus* is not prone to much fragmentation whereas *Quercus* and *Fraxinus* produce a lot of fragments. Between these two extremes, *Fagus*, *P. pinaster*, *Acer* and *Betula* display little fragmentation, whereas *Corylus* and *Carpinus* tend to be highly fragmented. *P. sylvestris* shows an intermediate response to fragmentation (Table 4 and Fig. 5c).

- However, interspecific variability is significant for the small fragments [1–2 mm], but less so for larger fragments [2–4 mm]. In the largest class size (>4 mm), only *Quercus* is different from the other taxa, producing a lot more fragments (Fig. 8).
- Wood with a marked porous zone, with large vessels, and with long radial file vessels (*Carpinus* and *Corylus*) produces the most fragments. Homogeneous wood (small isolated diffuse vessels or homoxylates) such as Populus, *Acer*, *Betula* or *P. pinaster*, *P. sylvestris* are those that produce the least fragments (Fig. 9).
- The presence of spiral thickenings (*Corylus*, *Carpinus* and *Acer*) is linked to a higher crushing strength. The low value of crushing strength is either linked to the porosity of the charcoal (*Fraxinus* and *Quercus)*, or to its low density (*Populus*, *P. sylvestris* and *P. pinaster).* In all cases, wood with a low density presents a low value for crushing strength. On the other hand, dense wood alternatively yields either very high or very low values (Fig. 9).

5. Interpretation

5.1. Focus on factors impacting fragmentation

- Density

The density of dry wood seems to have an impact on fragmentation: charcoal from dense wood is very fragmented while low to medium density wood charcoal presents low to medium fragmentation. Despite a low coefficient of determination (0.267), there is a significant positive correlation (Rhô = 0.517) between "wood density" (and therefore charcoal density) and the "total number of fragments". As the majority of the fragments are in the [1–2 mm] fraction, density is also logically correlated with the number of small fragments, as shown by the MCA analysis: the denser the wood, the higher the number of small fragments.

- Wood porosity vs. homogeneity

Fragmentation intensity is related to charcoal porosity. Cellular organization seems to be strongly linked to the fragmentation processes. Marked porous zones with large vessels or radial pore files are prone to fragmentation. Conversely, more homogeneous porous wood (with small isolated diffuse vessels or homoxylates) such as *Populus*, *Acer*, *Betula* or *P. pinaster*, *P. sylvestris* tends to be less fragmented.

- Length and width of the rays

The production of large fragments, as for *Quercus*, is also linked to the presence of a significant porous zone. But *Quercus* differs from the other species by the presence of multiseriate rays, which create fragile zones after combustion and account for the specific fragmentation mode of this species. For the other species, the rays, whether multiseriate or uniseriate, play a secondary role in both fragmentation and the mechanical properties (*cf.* central position and weak contribution in the construction of the MCA graphic).

5.2. Factors affecting crushing strength

In all cases, charcoal with a low density presents a low value for crushing strength, but high density charcoal alternatively yields very high or very low values. The relationship between charcoal density and crushing strength is thus not clear.

Corylus, *Carpinus* and *Acer* present a higher crushing strength than the other taxa. This characteristic is linked to the presence of spiral thickenings, which are preserved after charring. Conversely, the weak value of crushing strength is either linked to the porosity of the charcoal, in the case of *Fraxinus* and *Quercus*, or to its low density, as is the case for *Populus*, *P. sylvestris* and *P. pinaster*.

For dry wood, crushing strength is more or less proportional to wood density. Although *Quercus* is the wood with the highest density, its charcoal has the lowest crushing strength. This is due to the fact that charring induces the highest density loss for this species, perhaps because of its chemical composition. In addition, *Quercus* displays two orthogonal arrays of weak zones: an initial porous zone in a tangential direction and very large rays in the radial direction. This should be conducive to the initiation of a large number of cracks at rather low stress levels.

On the other hand, *Corylus*, *Carpinus* and *Acer* present a higher crushing strength than the other taxa, although the density of these species is no higher than that of *Fraxinus*, for example. They are more homogeneous woods with less distinct weak zones prone to fractures. Furthermore, the presence of spiral thickenings in the fibers, which are preserved after charring, appears to reinforce the structure of these charred woods.

Populus is characterized by a very homogeneous structure, but with a much lower density than the former species.

The low resistance of *Fraxinus* and *Fagus* charcoal, in spite of the rather high density of these woods, can also be explained by the presence of weak zones: tangential (initial porous zone) for *Fraxinus* and radial (large rays) for *Fagus*.

6. Discussion: from experimentation to the archaeological context

This study shows that the anatomical structure of the different tested wood taxa has a significant impact on the mechanical properties of charcoal. Charring produces less heterogeneous material than the different initial woods, but it does not erase the differences in density, organization and cellular composition, etc.

Thermal response, resistance to pressure, fragmentation and its modalities are largely dependent on the anatomical features and thus, on the physical characteristics of wood. Some of the noted differences are significant from an archaeological viewpoint. The alteration of the mechanical properties follows a differential thermal pattern depending on the species, which can cause differences in taxa fragmentation in the same hearth or among scattered charcoals. The formation temperature of charcoal also influences the fragmentation of the material (Théry-Parisot, 2013). Yet, the temperatures are highly fluctuant in a same combustion structure. It is therefore very difficult to archaeologically assess the question of the weakness of charcoal in relation to charring temperatures.

Fragmentation differences are mainly visible outside the [>4 mm] class size. These first results tend to show that anthracoanalysis limited to charcoal over 4 mm would induce less risk of under or over-representation of the different taxa, with the exception of *Quercus*, which is over-represented in this class size.

We also noted variations in crushing strength values from one species to another. This signifies that the rupture of the material is not initiated at the same pressure levels. Thus, from an archaeological perspective, in the same burial conditions, taxa will not necessarily demonstrate the same resistance capacity, which could

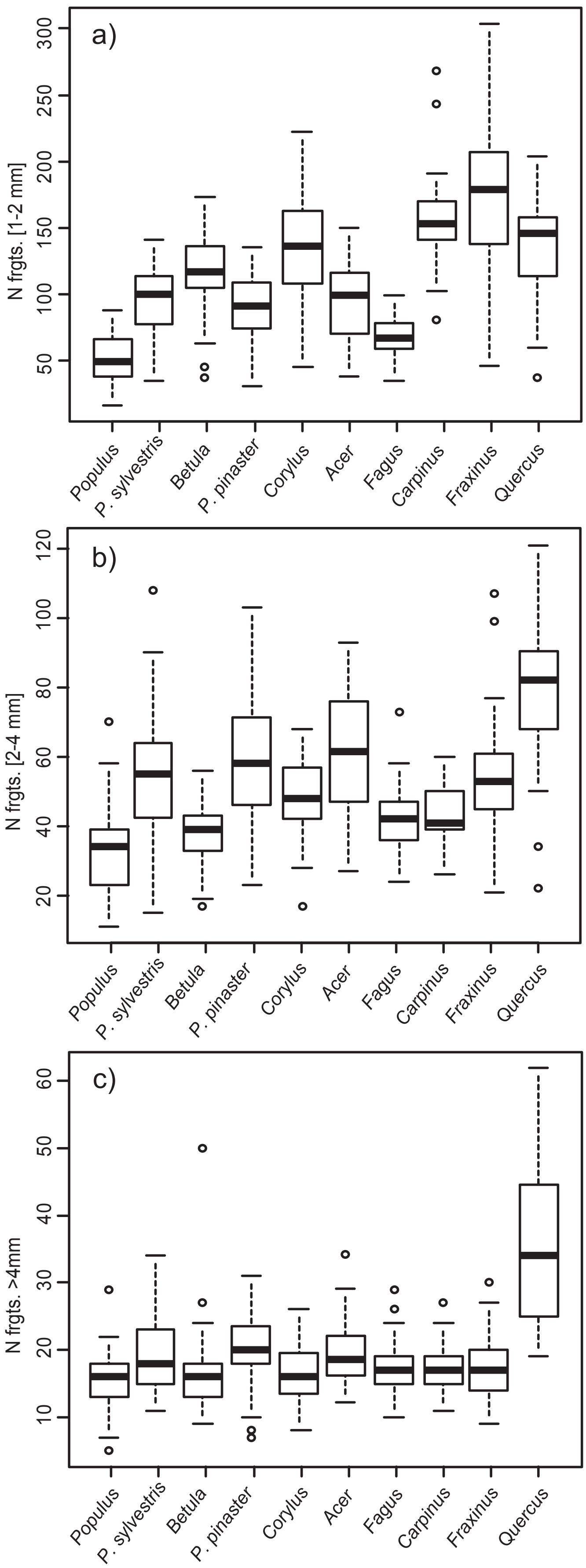

Fig. 8. Box & Whiskers plot for the number of fragments by species in the three class sizes: 1–2 mm (a); 2–4 mm (b); >4 mm (c).

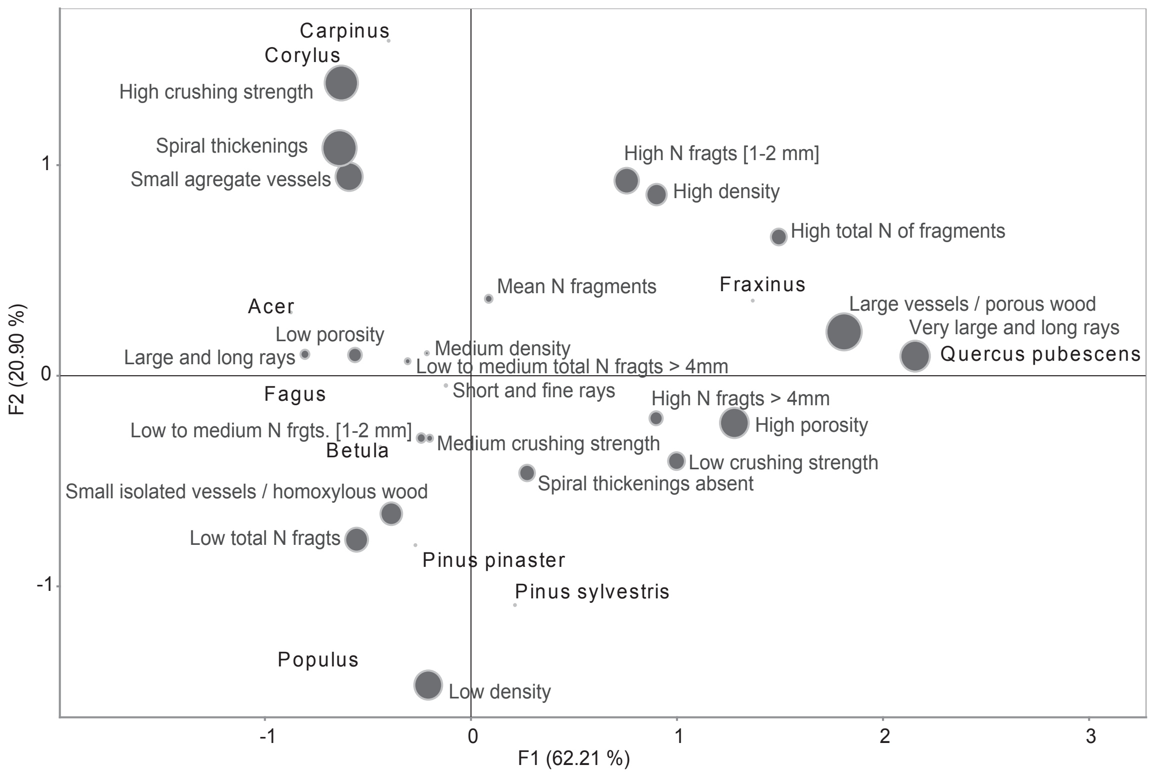

Fig. 9. Multiple Correspondence Analysis (symmetric graph): mechanical properties/ wood anatomy.

bias interpretation. However, crushing strength depends on the formation temperature of charcoal and has no impact on the quantity of fragments produced after the rupture of the material. This results in a set of extremely variable situations where it is impossible to assess or to measure all the parameters. It is thus important to study and to take account of burial conditions (the nature and intensity of post-depositional processes involved in the deposit formation, rate of burial at the scale of the site, but also intra-site variations), but it is nonetheless impossible to infer the mechanical behaviour of charcoal. However, when interpreting the data, it is imperative to bear in mind that some taxa are more fragile than others.

Conversely, the values obtained for crushing strength appear to be more significant. Charcoal tolerates relatively high pressure before fragmentation, oscillating between 8 and 19.4 MPa, with an average of 13.7 MPa. Mechanically, charcoal is considered to be fragile. In comparison, the crushing strength of common conifers (uncharred) is between 18 and 30 MPa, that of ordinary concrete is between 16 and 40 MPa and that of steel between 235 and 350 MPa (Ashby, 2005). Yet most post-depositional processes do not generate very strong pressure. Cattle exert greater static pressure (160–192 kPa) on soil than sheep (83 kPa), although this pressure is at least doubled when animals are walking (Drewry, 2006), which represents less than 0.4 MPa. The value for human beings cannot be much higher. The maximum pressure measured during freeze propagation experiments in a limestone notch is about 6 MPa (Bost, 2008).

Thus, the intense fragmentation of charcoal (extending at times to the disappearance of the macroscopic fraction), characteristic of some Pleistocene sites (Théry-Parisot, 2001; Théry-Parisot et al., 2010a; Beresford-Jones et al., 2010, Marquer et al., 2012) does not seem to result from single, short-term processes. However, classic processes, such as freeze-thaw action or trampling occurring repeatedly during the course of a phase of low (or no) sedimentation, could doubtlessly generate progressive charcoal weakening, leading to extreme fragmentation. Under natural conditions, the repetition of these processes induces progressive mechanical fatigue of the material (which is not measured in our experiment), thereby increasing charcoal damage. Finally, it is important to distinguish post-depositional processes from fuel management. The disappearance of the coarse fraction due to burial conditions sometimes leads to the misinterpretation of charcoal scarcity, suggesting that other fuels, such as bones or dung, were used. Further investigations based on the study of very thin coarse fractions combined with geoarchaeological studies, are required, so that more reliable interpretations can be proposed. (Fernández-Jalvo et al., 2010; Marquer et al., 2010, 2012; Miller et al., 2010; Scott and Damblon, 2010).

Naturally, these observations must be moderated, notably because of the way the samples are made, which is quite far removed from the archaeological reality. Complementary analyses are required, incorporating other criteria, such as the chemical composition of the taxa. Moreover, this paper does not take account of the other stages of fragmentation, namely combustion. Through cross indexing data from research on combustion and post-depositional processes, it will become possible to gain a better understanding of anthracological assemblages.

Based on an innovative experimental approach combining both archaeobotany and biomechanics, these results are fundamental for a comprehensive understanding of archaeology, archaeobotany and palaeoecology. They document the mechanical behaviour of charcoal, a largely unknown domain up until now. They enhance our understanding of charcoal taphonomy by providing added resolution and improving the accuracy of charcoal analysis.

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